



## REPLACEMENT SHEET

### *Fig. 1A*

#### Step 1

##### Preparation of a silver nitrate solution (solution A)

A solution of 2.1 g silver nitrate in 1 ml water is adjusted to 500 ml with isopropanol.

#### Step 2

##### Preparation of a sodium acetate solution (solution B)

A solution of 0.4 g sodium acetate in 0.5 ml water is adjusted to 500 ml with isopropanol.

##### Preparation of a sulfur capturing liquid

#### Step 4

Solution A is mixed with solution B at ordinary temperature, and a white suspended matter (silver acetate) is formed.

##### Sulfur capturing liquid purification operation (No. 1)

#### Step 5

The white suspended matter is filtered off with a millipore filter (0.45  $\mu$ m) and is removed.

##### Desulfurization with X-rays

By irradiation with X-rays (X-rays containing the absorption edge wavelength (5.018  $\text{\AA}$ ) of sulfur), sulfur contained as the impurity is changed into a silver compound containing silver sulfide. The time of irradiation with X-rays is 1 hour (for 15 ml container). The sample is then left.



## REPLACEMENT SHEET

### *Fig. 1B*

#### Step 6

Sulfur capturing liquid purification operation (No. 2)



The black precipitates are filtered off through a millipore filter (0.45  $\mu$ m) and sulfur contained as the impurity is removed as silver compound (mainly containing silver sulfide).

#### Step 7

Dissolved oxygen removing operation



#### Step 8

The liquid is bubbled with  $N_2$  for 20 minutes, to remove dissolved oxygen.

#### Step 9

Sulfur capturing liquid stabilization operation



10 ml benzaldehyde is added to the liquid, to reduce oxidation of the liquid.

#### Step 9

Storage of the sulfur capturing liquid

The liquid is stored in a cool and dark place.



## REPLACEMENT SHEET

*Fig. 2*

Step 1

Dissolved oxygen removing operation

Step 2

The liquid is bubbled with N<sub>2</sub> for 20 minutes, to remove dissolved oxygen.

Sample collection

Step 3

5 ml sample is collected in the liquid sample container

Sulfur capturing liquid collection

Step 4

2 ml is placed in the sample-containing container and stirred sufficiently

Preliminary reaction of the sample

Step 5

By irradiation with X-rays (X-rays containing the absorption edge wavelength (5.018 Å) of sulfur), a very small amount of sulfur in the sample is converted into a silver compound (mainly containing silver sulfide). Silver reacting elements other than sulfur are also converted into silver compounds. These are also co-precipitated with silver in the bottom of the measurement container. The time of irradiation with X-rays shall be 30 minutes/sample

Extraction of the object component (silver compound)

In the presence of co-sedimented silver compounds and silver, scattered X-rays are increased and the absorption of a characteristic X-ray of S occurs, thus making analysis of a very small amount of sulfur difficult. Accordingly, ammonia or aldehyde is added to the sample and then left at a constant temperature (30 °C) for about 20 hours. By this operation, silver compounds and silver other than the object component are dissolved, diffused and removed from the sample measurement surface.